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Calcium Oxide Derived from Okpella Limestone as a Heterogeneous Catalyst for Biodiesel Production: Synthesis, Characterisation and Stability Assessment

Abdulrahman S. USMAN[1*], Folorunsho ABERUAGBA[2], Moses A. OLUTOYE[3], Umar MUSA[4], Mohammed ALHASSAN[5]

[1*,2,3,4,5]Department of Chemical Engineering, Federal University of Technology, Minna, Nigeria

[1*]uabdurrahman21@gmail.com, [2]f.aberuagba@futminna.edu.ng, [3]m.olutoye@futminna.edu.ng, [4]umar.musa@futminna.edu.ng, [5]moh.alhass@futminna.edu.ng

Abstract

Sustainable energy alternatives have increased the pace of the research on low-cost and low-environmental-impact catalysts used in the production of biodiesel. This paper examines the synthesis, characterisation, and performance of calcium oxide (CaO) catalysts produced from Okpella limestone, which is in Edo State, Nigeria. This was done by calcining the limestone at 900°C over a period of five hours to break down the calcium carbonate ($CaCO_3$) into catalytically active CaO. FTIR, XRD, XRF, SEM, BET, and TGA methods were used to characterise the raw and the calcined materials to assess their physicochemical characteristics and their appropriateness to the production of biodiesel. The XRD and the FTIR analyses verified the conversion of calcite into phase-pure CaO with high crystallinity. XRF and EDS data revealed that it contained 89.23 wt.% of CaO, which signified high purity and high basicity. SEM micrographs showed a porous irregular morphology, and BET analysis showed mesoporosity with a high surface area of 61.21 m^2/g, a pore diameter of 3.14 nm and a pore volume of 0.11 cm^3/g, which are significant characteristics for efficient transesterification. Complete decomposition of $CaCO_3$ at 650°C to 800°C was confirmed by thermogravimetric analysis. Catalytic stability was shown to be six reaction cycles, with reusability tests showing biodiesel yields of over 90% before deactivation occurred gradually due to surface fouling and leaching. These results make Okpella limestone a viable, low-cost, and locally available raw material in the manufacture of high-grade CaO catalysts. The research advocates the production of cleaner biodiesels, and it helps Nigeria shift into renewable energy systems.

Keywords: *Calcium oxide, Okpella limestone, biodiesel, heterogeneous catalysis, renewable energy, transesterification, Nigeria.*

1.0 Introduction

The accelerating global energy crisis has intensified efforts to transition toward renewable and sustainable energy sources. This is mainly due to the expedited population growth and urbanisation [1],[2]. The need to find renewable and sustainable energy sources has become an urgent concern due to their increasing demand and the challenges of climate change [3]. Biodiesel has become one of the alternatives among other biofuels produced majorly by transesterification of vegetable oils or animal fats with alcohol in the presence of a catalyst [4]. It is biodegradable and non-toxic, and it generates much less greenhouse gas emissions in comparison with conventional diesel [5],[6]. Nonetheless, large-scale biodiesel production continues to be problematic because of high costs of production, low catalyst activity, catalyst recovery and stability issues [6],[7].

Biodiesel production is greatly enhanced by heterogeneous catalysis, offering easier separation of catalyst and product, catalyst reuse, and less wastewater generation, with fewer environmental concerns when compared to homogeneous catalysts such as NaOH and KOH [8]. Calcium oxide (CaO) and its derivatives are the most predominant heterogeneous catalysts in the production of biodiesel because of their high basicity, relative abundance, low cost, and high catalytic activity under moderate transesterification conditions [9]. Calcium oxide can be synthesised from various sources, including eggshells, animal bones, seashells, snail shells, and limestone. Among these, limestone remains the primary source of calcium oxide [5].

Limestone is composed mainly of calcium carbonate ($CaCO_3$). It is a naturally abundant precursor for CaO production. Its chemical composition makes it an accessible and cost-effective raw material for CaO synthesis, especially in regions where limestone deposits are readily available [10],[11]. Calcination is the process by which limestone is heated to a high temperature, resulting in the decomposition of $CaCO_3$ into calcium oxide (CaO) and carbon dioxide (CO_2) [12],[13].

Okpella limestone is a specific variety indigenous to Okpella, located in the northern part of Edo State, southern Nigeria. It lies between latitudes $7^0$14'17.80"N to $7^0$24' 6.97"N, and longitudes $6^0$12'36.60"E to $6^0$29'31.57"E. The limestone is relevant due to its unique mineralogical composition, lower impurity levels, and economic accessibility. [14] Its use as a feedstock for the synthesis of CaO catalysts presents a viable option to

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leverage local mineral resources, reduce production costs, and promote regional sustainable development. However, the use of CaO catalyst in biodiesel catalysis is not without challenges. These are particularly related to catalyst stability, deactivation, and leaching during reaction cycles [15]. Catalyst deactivation mechanisms limit the reusability and long-term efficiency of CaO catalysts. Such mechanisms include sintering, leaching of active species, and surface fouling [16], [17]. Therefore, understanding and enhancing the physicochemical stability of CaO derived from Okpella limestone in biodiesel production is critical to practical and sustainable applications.

Recent research has increasingly focused on improving CaO catalyst performance by focusing on mitigation techniques for the deactivation challenges. These approaches include doping with promoters, surface modification, and support integration [18]. Also, innovative synthesis methods such as calcination at controlled conditions, nanoparticle engineering, and the use of mixed oxides have reported promising gains in catalytic activity, stability, and reusability [15],[19].

Despite these significant strides, there remains a notable research gap. This gap concerns the valorisation of locally sourced limestone deposits like Okpella limestone for CaO catalyst production. Previous studies have shown potential in using commercial CaO catalysts; however, the specific characteristics, stability behaviours, and performance metrics of Okpella limestone-based catalysts are underexplored. Such knowledge is very crucial to the development of region-specific sustainable technologies that capitalise on available mineral resources.

This study aims to bridge this gap by systematically investigating the production, physicochemical characterisation, and stability behaviour of CaO synthesised from Okpella limestone.

2.0 Methodology

2.1 Catalyst Preparation and Characterisation

The limestone used for this research was obtained from the BUA quarry in Okpella, Edo State, Nigeria. The limestone was first washed with deionized water, dried in an oven at a temperature of 120℃ for four hours. Then crushed and sieved with 1.5 mm sieve to obtain a fine powder. The finely ground limestone powder was soaked in a diluted hydrochloric acid followed by another drying in oven for about three hours. The sample was then subjected to a high-temperature calcination process at 900°C for 5 hours in a muffle furnace [13]. This induced the conversion of the limestone (predominantly calcium carbonate) into calcium oxide, which is the active catalytic component responsible for facilitating the transesterification reaction between the neem seed oil and alcohol [20],[21].

Characterisation of the raw limestone and the catalyst

The raw limestone and prepared catalyst was characterised using various analytical techniques. This was carried out to study the catalysts and its material crystalline structure, chemical composition, surface morphology, and thermal stability [21],[22]. The following are the characterisation techniques employed:

Furrier Transforms Infrared Spectrophotometer

The infra-red spectrometer analysis of calcium oxide catalyst was performed using Bruker Alpha II infra-red spectrometer. In carrying out the analysis, the spectrometer was powered and allowed to warm up for 5 minutes. The spectrometer sample press tip and the diamond sampling window were cleaned and made clear of any residue from previous sample. The sample was then placed on the cleaned crystal window and the overhead press tip adjusted until it exerted the desired pressure on the introduced sample. Transmission spectra of composite films were recorded at ambient temperature. Sample was scanned from 4000 to 500 cm-1 with resolution of 0.4 cm^{-1}.

X-Ray Diffraction Analysis

XRD characterisation of thin film samples was done at Umaru Musa Yar'adua University, Katsina State, Nigeria, in the Central Research Laboratory with the X-ray Diffractometer Thermo Scientific model ARL'XTRA X-ray and serial number 197492086.

The X-ray diffraction analysis was conducted to determine the crystalline structure of the raw limestone and the catalyst. Powdered samples were evenly distributed on a clean glass slide, and the diffraction patterns were recorded within a 2θ range of 10–80°. The identified crystalline phases were determined by comparing the observed d-spacing values and peak positions with the reference data from the Joint Committee on Powder Diffraction Standards (JCPDS) database.

X-Ray Fluorescence (XRF) Analysis.

The elemental and chemical composition of calcium oxide (CaO) was analysed with a Thermo Fisher Scientific XRF analyser in Switzerland. Model ARL.QUANT X EDXRF and serial number 9952120. The dried powder was weighed and formed into a pellet by compression in a hydraulic press with an appropriate binder (boric acid). The XRF spectrometer was calibrated by using certified reference material that had a known oxide composition akin to the sample. The sample holder was loaded with the pellet, and the analysis was done using an operating condition of 30 kV and 40 uA with a rhodium (Rh) anode X-ray tube. The secondary X-rays emitted were identified and processed with the help of basic parameter correction algorithms to determine the concentration of major oxides.

Brunauer-Emmett-Teller (BET) analysis

This analysis was used to determine the specific surface area of the catalyst. This is crucial in understanding the effectiveness of adsorption processes, as catalysts with higher surface area generally have more sites available for adsorption/reaction. The BET analysis is valuable in characterising the porosity and surface properties of the catalysts. It provides insights into the efficiency of these materials in adsorption process.

Scanning Electron Microscopy (SEM) Analysis

The surface morphology of the prepared limestone-derived catalyst was examined using Scanning Electron microscope model: PRO:X: 800-07334 Phenom World and serial number MVE01570775. The catalyst sample of 0.05mg was dispersed onto carbon adhesive tape and then coatted with a thin layer of gold-palladium using a sputter coater for around 5 minutes to enhance the conductivity and improve the image quality. The SEM instrument was operated at an accelerating voltage of 5 kV to capture high-resolution images that provided detailed information about the catalyst surface morphology and particle characteristics.

2.2 Catalyst Reusability Studies

The performance, reusability and long-term stability of the prepared limestone-derived catalyst are crucial aspects of its practical application. After the transesterification reaction using neem seed oil, the catalyst was recovered, washed with n-hexane to remove any adhering biodiesel, and then dried at 110°C for 2 hours. The dried catalyst was reused in subsequent transesterification runs to evaluate its stability and performance over multiple cycles.

3.0 Results and Discussion

3.1 Characterisation of limestone

Fourier transform infrared (FTIR) analysis

The FTIR spectrum of the limestone sample shown in Fig. 1 displays characteristic vibrational features consistent with calcium carbonate ($CaCO_3$) as the primary phase, which is the major constituent of natural limestone.

The intense absorption bands around the 1400–1500 cm^{-1} region are attributed to the asymmetric stretching vibrations of the carbonate ion (CO_3^{2-}). This confirms the presence of calcite, the most common crystalline form of calcium carbonate found in limestone [9],[23]. The absorption near 875 cm^{-1} corresponds to the out-of-plane bending modes of the carbonate group, further supporting the identification of the calcium carbonate phase [24]. Additionally, a sharp band near 700 cm^{-1} can be attributed to the in-plane bending of the carbonate ion, which is characteristic of calcite [6]. The absence of substantial bands related to other mineral impurities or organic residues suggests that the limestone sample is relatively pure, which is advantageous for catalytic applications following calcination.

The broad absorption band between 3400 and 3600 cm^{-1} is due to O–H stretching vibrations and indicates trace amounts of adsorbed moisture or hygroscopic water molecules on the limestone surface. This is typically observed in natural mineral samples exposed to humidity [25].

Understanding the initial mineralogy and purity of the limestone through FTIR analysis is crucial, as the presence of impurities, organic matter, or mixed carbonate phases can influence the efficiency of subsequent calcination, catalyst properties, and catalytic performance [6],[19].

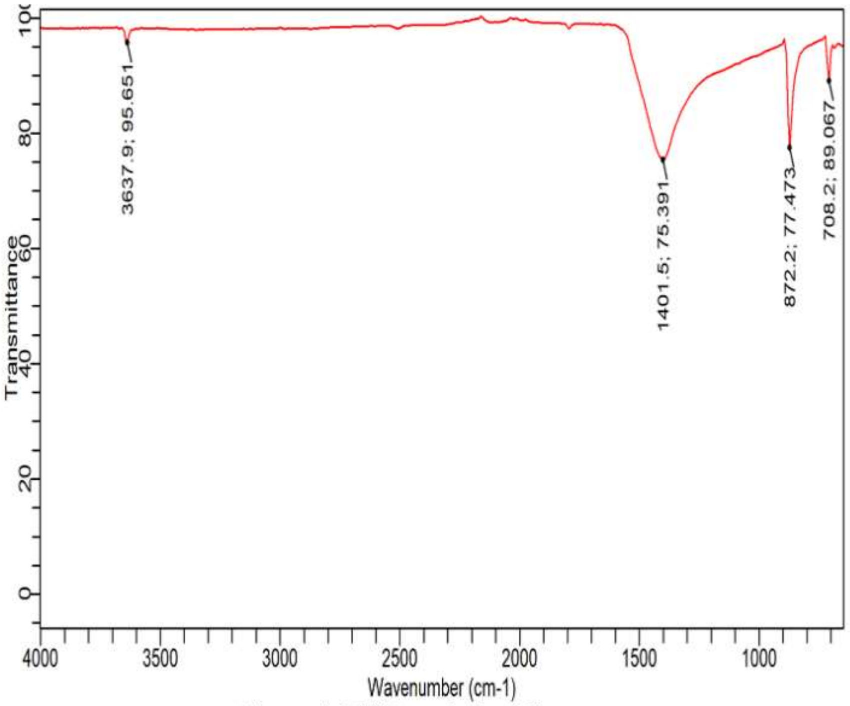

Figure 1: FTIR analysis of limestone.

Thermogravimetric analysis of limestone

TGA and DTA of limestone indicated that a large weight loss occurs at a temperature range of 650°C to 800°C, which is associated with the thermal decomposition of calcium carbonate ($CaCO_3$) into calcium oxide (CaO) and carbon dioxide (CO_2). The calcination of limestone for this work was carried out by heating to 900°C and a five-hour isothermal treatment at 900°C, as evident in the TGA plot in Figure 2. This calibration guarantees the full decomposition of $CaCO_3$ to highly crystalline CaO and minimises residual carbonate phases [13]. Several studies [26] pointed out that full conversion of calcium carbonate from natural sources requires temperatures beyond 800°C and that crystallinity as well as phase purity increase when held at high temperatures. Similarly, [27],[28] claimed that natural-origin CaO catalysts calcined at 900°C have high reusability and stability across several reaction cycles, which confirmed the efficiency of the thermal treatment regime used in this study.

The lack of a prominent mass loss below 200°C in the TGA plot also shows that the moisture and volatile content of the limestone are low, indicating a high purity of the precursors and successful drying during pre-calcination treatment. The single-event decomposition profile in both TGA and DTA indicates little to no contamination by silicate or aluminium phases, which would otherwise broaden or shift the thermal transitions.

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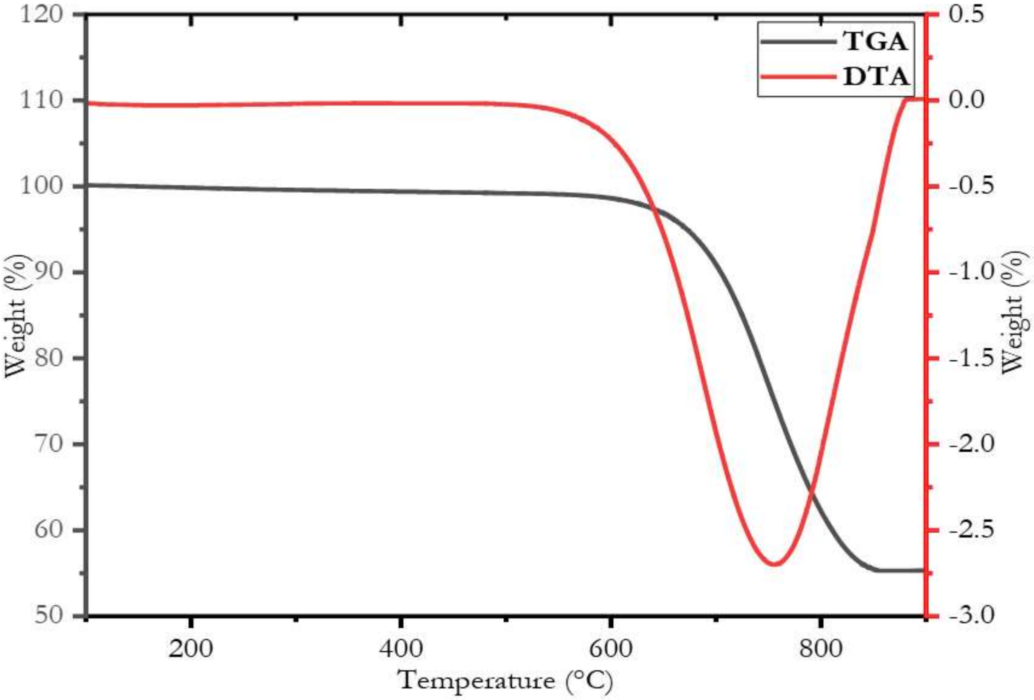

Figure 2: TGA and DTA of limestone

XRD analysis of limestone

The XRD pattern of untreated limestone at Okpella, Edo State, Nigeria, depicts that it contains high-purity calcite ($CaCO_3$), as it has prominent peaks at 2θ values of about 29.4°, 39.5°, 43.2°, 47.5°, 48.6°, 56.6°, and 58.9°. These significant reflections are those of the (104), (113), (202), (018), (116), (214), and (300) crystallographic planes of calcite, which have been confirmed by standard JCPDS reference files. These high relative intensity peaks, especially the dominant peak that is at 29.4°, reflect a well-crystallised calcite phase. The profile is closely related to the results found by [29], who observed the same XRD patterns of high-grade calcite limestone with minor impurities.

The lack of secondary minerals, such as quartz (SiO_2), dolomite ($CaMg(CO_3)_2$) or clay minerals, is another valuable indication of the mineralogical purity of the limestone, a significant feature for application as catalysts. The peaks of calcite are dominant and sharp, and the peak profile is well-defined, indicating that thermal decomposition will produce highly reactive calcium oxide (CaO) with minimal contamination that may affect the performance of the catalyst.

The XRD analysis evidently underpins the appropriateness of this Okpella limestone to produce high-purity CaO and its subsequent use as a practical biodiesel catalyst.

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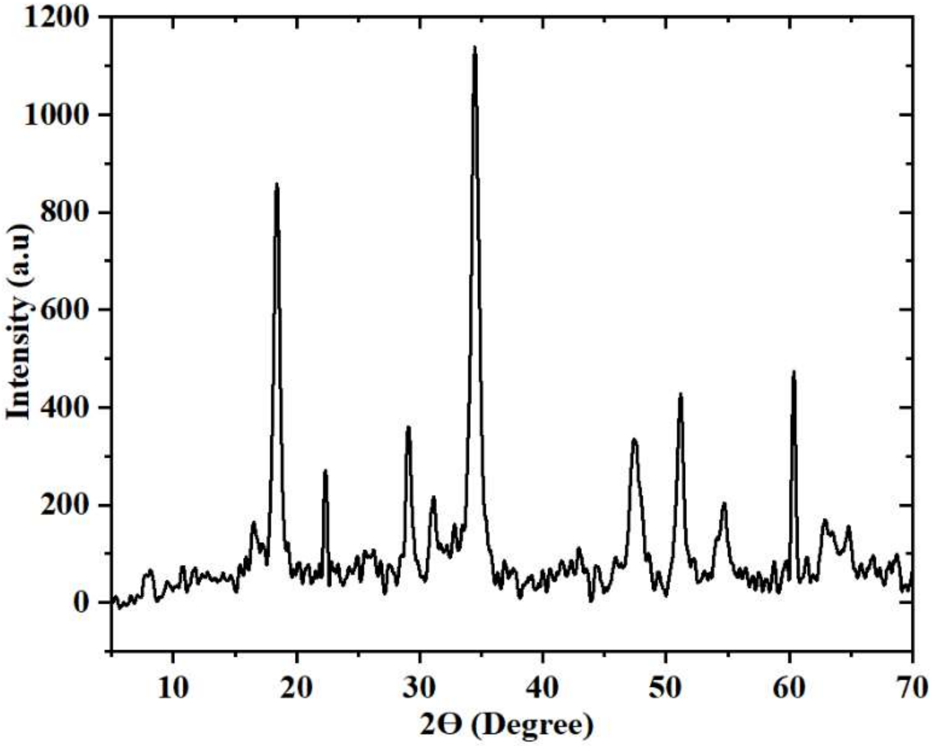

Figure 3: XRD Analysis of Limestone.

3.2 Characterisation of the Catalyst

Catalyst major component phase

The X-ray diffraction (XRD) pattern (Fig. 6) of the synthesised calcium oxide (CaO) catalyst is shown in Figure 4. In contrast to the XRD pattern of raw limestone (Fig. 3), after calcination, the pattern changes completely, with all calcite peaks disappearing and new intense reflections emerging at approximately 32°, 37°, 53°, 64°, and 67°, which correspond to the cubic lattice planes of calcium oxide (CaO). This shift confirms the successful thermal decomposition of $CaCO_3$ to CaO, as the transformation from a rhombohedral calcite structure to a cubic CaO structure results in a distinct set of diffraction peaks. These peaks also correspond with Joint Committee on Powder Diffraction Standards (JCPDS) files for cubic CaO. This result is consistent with findings of [30], who reported similar XRD profiles for CaO derived from limestone and agricultural waste materials such as eggshells and animal bones. The studies emphasised that well-crystallised CaO is critical for effective transesterification and that peak purity is closely linked to the calcination temperature. Additionally, [31] observed that CaO prepared from mussel shells shows nearly identical diffraction patterns to commercial CaO, highlighting the reproducibility and effectiveness of CaO synthesis from various natural precursors.

The absence of significant amorphous humps or secondary phases, such as unreacted $CaCO_3$ or $Ca(OH)_2$, in the current pattern suggests high phase purity and structural stability. This aligns with observations of [32], noting that the presence of moisture or insufficient calcination time often results in residual carbonate peaks. Therefore, the XRD results strongly support the successful preparation of phase-pure, crystalline CaO suitable for heterogeneous catalysis in biodiesel production.

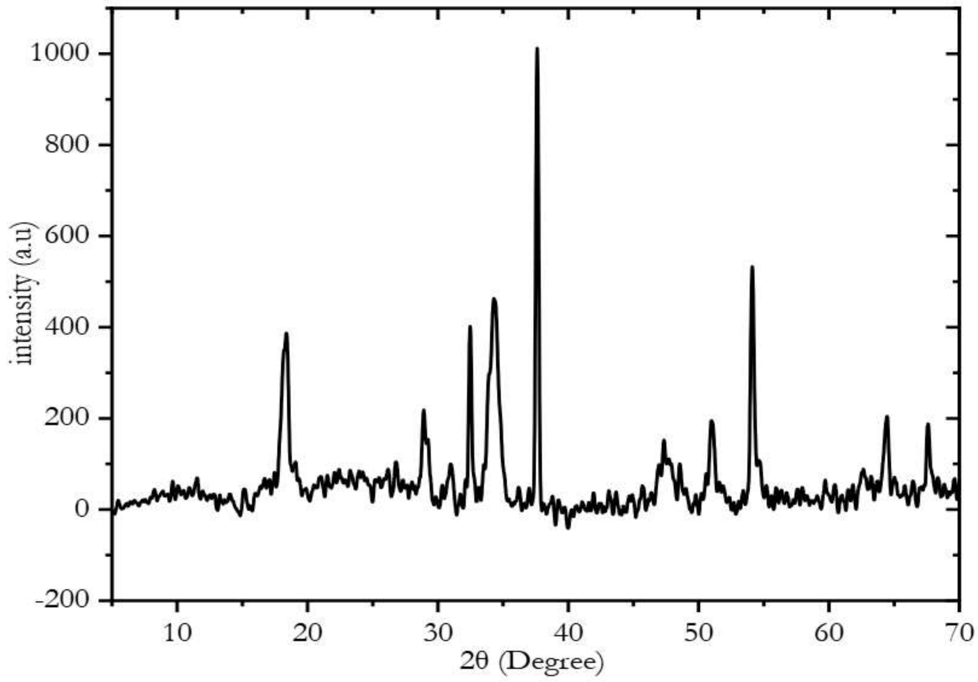

Figure 4: XRD Analysis of Calcium Oxide Catalyst

Catalyst composition

The synthesised catalyst's elemental and chemical makeup, as displayed in Table 1, reveals a high CaO content of 89.23wt.% (91.01 mol%), demonstrating the efficient thermal decomposition of limestone at high temperatures. This result closely aligns with findings by [31], that reported CaO contents of 86 wt.% in catalysts derived from marine limestone calcined at 900°C. Similarly, [33],[34] documented comparable levels of CaO compositions from natural sources such as oyster shells and eggshells, affirming that biogenic and geological sources can both yield highly pure CaO under optimal calcination conditions. The elemental analysis supports this assertion, with calcium comprising 63.77% of the total elemental composition, indicating a dominant CaO phase, essential for efficient base catalysis in transesterification reactions.

Minor components such as MgO, Al_2O_3, and SiO_2 are present in concentrations known to enhance catalyst performance. MgO contributes additional basicity and structural stability, while Al_2O_3 may improve surface area and mechanical durability. Although SiO_2 can form calcium silicates at elevated temperatureres, its low concentration poses minimal risk to active site availability. The presence of strontium is typical of limestone and may add further basic characteristics. These secondary oxides are frequently reported in catalysts derived from biogenic materials and are associated with improved resistance to deactivation [34],[35]. Trace elements such as Fe, Sn, and Cl reflect the geological origin of the limestone however are within acceptable limits for catalytic applications.

Overall, the composition suggests a cost-effective, sustainable alternative to synthetic catalysts, with the benefit of structural reinforcement from naturally occurring trace components.

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Table 1.1: Chemical and Elemental Composition of Catalyst Obtained Using XRF

S/N	Component	Concentration (wt.%)	Mole %
1.	CaO	89.23	91.01
2.	SO_3	0.81	0.58
3.	MgO	2.52	3.57
4.	Al_2O_3	4.36	2.44
5.	SnO_2	0.46	0.15
6.	SiO_2	0.95	0.91

S/N	Element	Concentration (%)
1.	Ca	63.77
2.	Al	2.31
3.	Mg	1.52
4.	Sr	0.54
5.	Sn	0.36
6.	Fe	0.27
7.	Si	0.45
8.	Cl	0.44
9.	O	29.86

Catalyst morphology

The SEM analysis (Figure 5) of the calcium oxide (CaO) catalyst shows the agglomeration of large, irregular particles with rough surfaces and apparent cracks. The morphology is related to the thermal decomposition of calcium carbonate ($CaCO_3$) in the calcination process. Fissures and granular texture indicate that the calcination procedure was successful in facilitating the production of a high-surface-area catalyst. The coarseness and porosity that are seen in this image are explained by the release of CO_2 gas as $CaCO_3$ decomposes, creating voids and enhancing the catalyst's surface area. [9] reported a similar microstructure of limestone-derived CaO calcined at elevated temperatures, with many cracks, which allows maintenance of catalytically active sites.

The morphological characteristics of this catalyst, which are supported by a random, porous, and cracked particle structure, are well known to intensify the basicity and reactivity of CaO used in transesterification reactions to produce biodiesel [5].

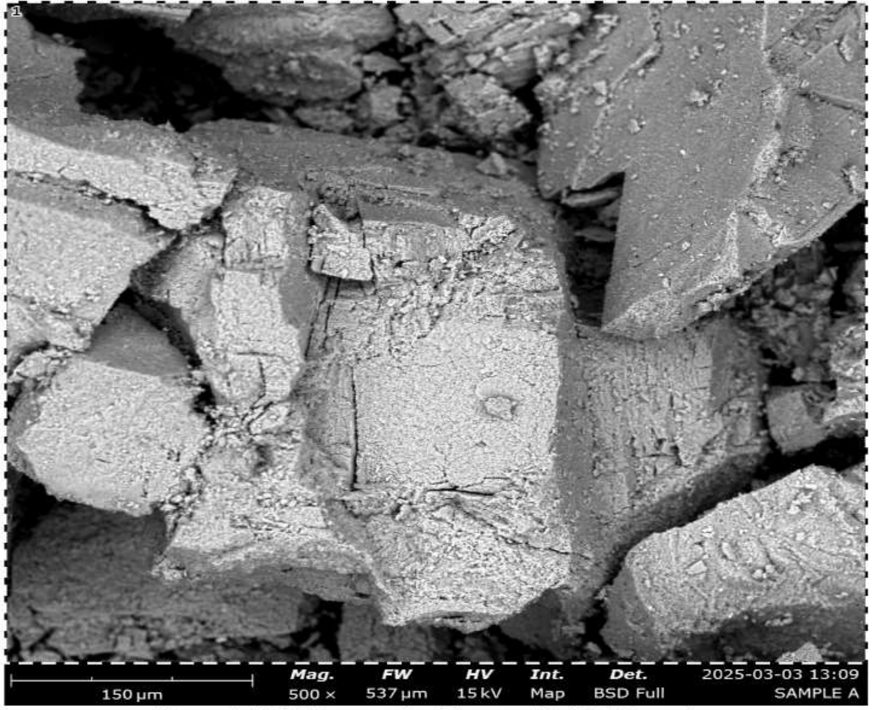

Figure 5: SEM image of calcium oxide (CaO) catalyst

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Energy dispersive X-ray spectroscopy (EDS) analysis

The spectrum in Figure 6 shows an energy dispersive X-ray (EDS) analysis of a calcined limestone sample. The peak that is predominant is that of calcium (Ca), which confirms that calcium carbonate ($CaCO_3$) was converted to calcium oxide (CaO) by the calcination. The trace elemental impurities that often occur in naturally derived limestone-based catalysts are indicated by the subsequent minor peaks of potassium (K), sodium (Na), phosphorus (P), chlorine (Cl), iron (Fe), titanium (Ti), and manganese (Mn).

This result is consistent with what has been reported in prior EDS analyses of limestone-derived catalysts that contain Ca compositions greater than 90% following calcination at temperatures above 800°C [9],[35]. It is not uncommon to find K and Na in calcined limestone; these alkali metals could be remnants of parent rock [10]. The lack of sharp peaks related to Si or Al means that there is little silicate or alumina contamination, which is beneficial since these additives may decrease catalyst basicity and reactivity [5].

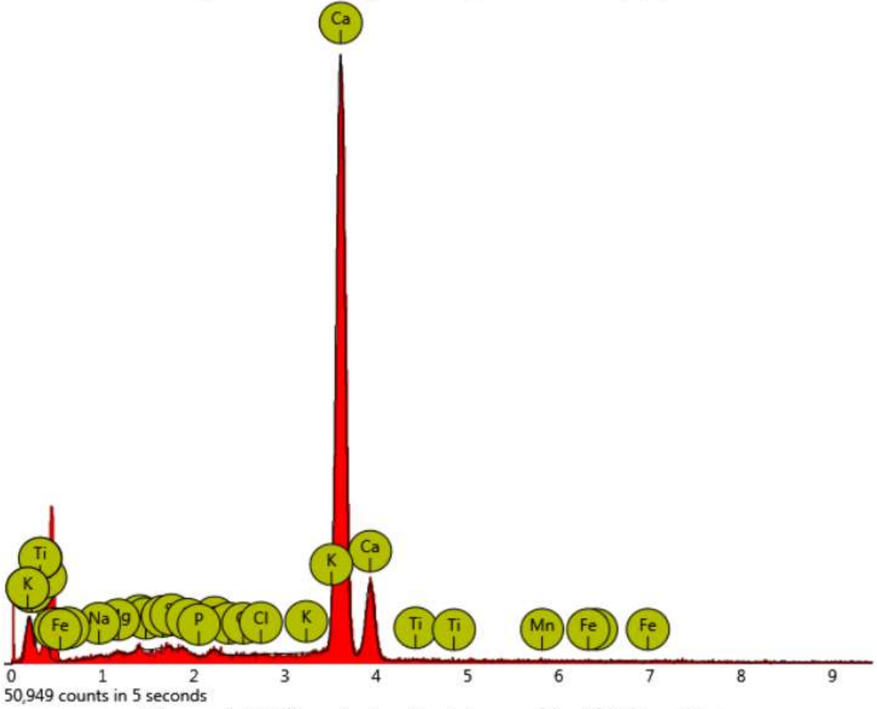

Figure 6: EDS analysis of calcium oxide (CaO) catalyst.

Catalyst structure, surface area, pore volume, and pore diameter

The BET (Brunauer Emmett Teller) analysis of the calcium oxide (CaO) catalyst is characterised by a large surface area, pore volume, and a relatively small pore diameter (Fig. 7). These results indicate that the catalyst has a large surface area and mesoporous structure; pore diameters, 2-50 nm, are typically mesoporous [36]. Mesoporosity is beneficial in catalytic applications due to enhanced reactant accessibility and diffusion and is also supported by the isotherm profile, which shows a strong hysteresis loop at larger relative pressures [13].

Comparative analysis reveals that the surface area in this study is higher than most of the literature on CaO catalysts prepared from natural sources. [37] lists a BET surface area of 34.7 m^2/g and a pore diameter of 3.12 nm for CaO obtained from limestone and a range of 25-60 m^2/g for CaO catalysts synthesised from animal bones and shells [38]. Conversely, [39] obtained 411 m^2/g surface area for a catalyst prepared from banana peel ash/LiCaO/$Fe_2(SO_4)_3$ blends. The increased surface area in this study may be due to an optimal calcination temperature and/or to the purity of limestone, which are well-known factors that have a large impact on textural characteristics [40]. The pore volume and pore diameter also fall in the range of the mesoporous regime. However, the smaller pore diameter indicates a higher concentration of active sites, a characteristic that enhances the catalytic activity of transesterification reactions [36].

The modern direction in catalyst development focuses on ensuring the maximum surface area and the pore structure management to achieve the higher efficiency and the stability of the catalyst, particularly in biodiesel synthesis from non-edible feedstocks [13],[40]. These trends are supported by the BET results of this study, in which it was determined that limestone-derived CaO can have better textural properties with the right processing.

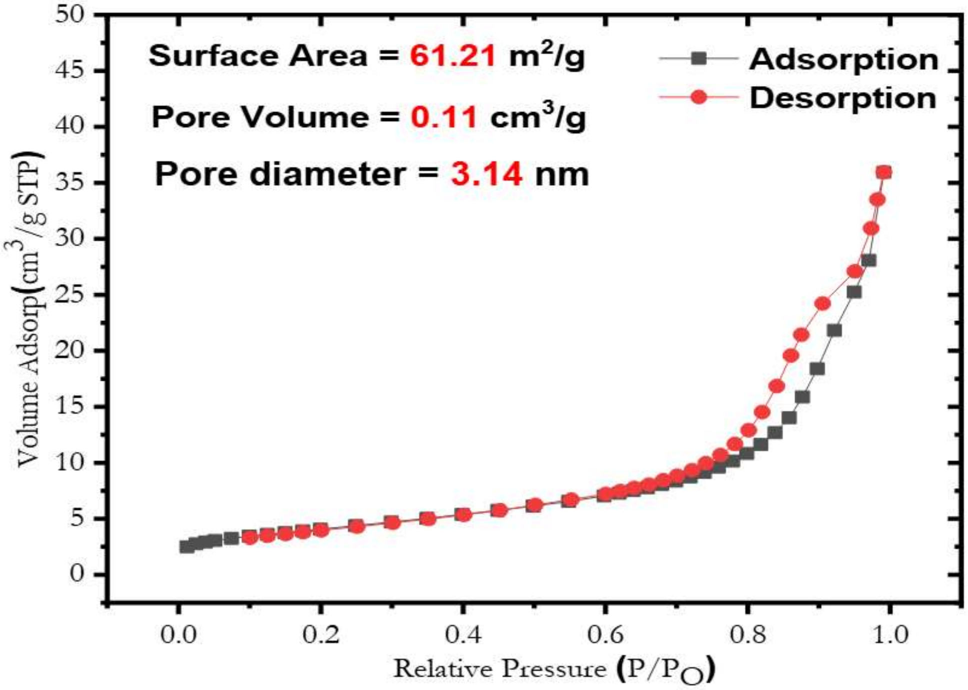

Figure 7: BET (Brunauer-Emmett-Teller) analysis of the calcium oxide (CaO) catalyst.

3.3 Catalyst Reusability

The reusability plot of the catalyst shown in Figure 8 indicates that the limestone-based calcium oxide (CaO) catalyst maintained a biodiesel yield of above 90% for six reuses when used to catalyse transesterification of neem seed oil. The catalytic activity decreases significantly after the sixth cycle, and the yield is reduced to 50% after the seventh cycle. This tendency shows that the catalyst CaO is effective at first. However, with reuse, its activity decreases greatly. The first stability may be explained by the fact that CaO is a basic substance, and its structure is stable, which allows transesterification reactions to occur efficiently in the early cycles [4].

This finding is in line with other research on the reusability of the CaO catalyst. A study by [15] noted that CaO obtained from limestone maintained more than 90% yield in up to 8 cycles of transesterification, with a significant decline in activity after this stage due to leaching and catalyst fouling. In the same way, [41] determined that the CaO catalysts could be reused 7-10 times before the biodiesel yield decreased significantly, which was explained by the formation of calcium glyceroxide, the deposition of organic impurities on the catalyst surface, and exhaustion of active CaO sites by reaction with water or CO_2 [42]. The trend observed in the current study therefore agrees well with the literature and validates the normal lifespan of reusability of CaO catalysts derived from limestone and other natural sources.

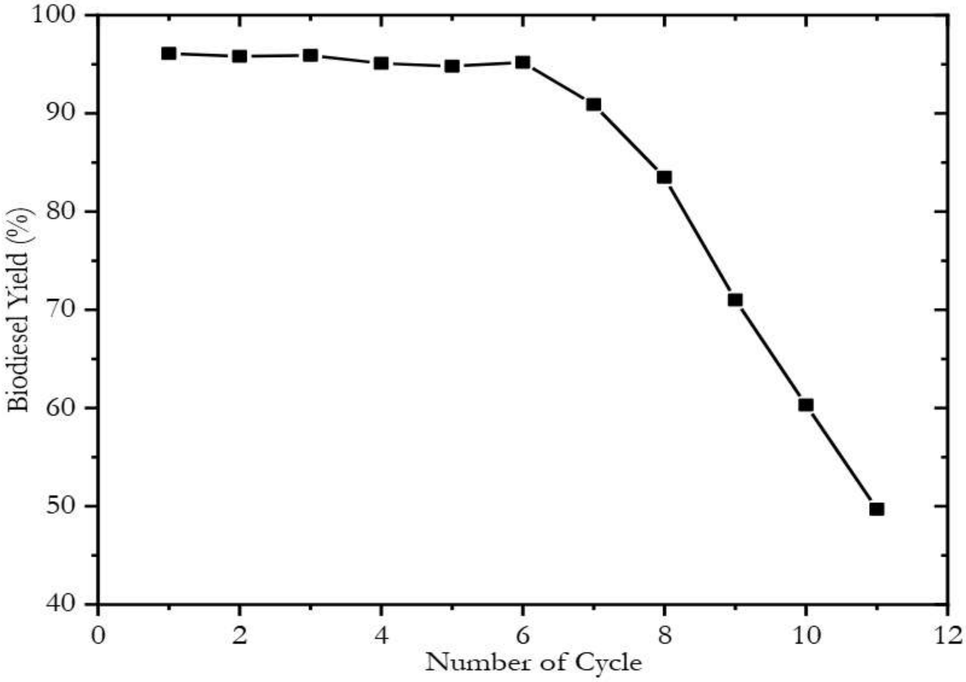

Figure 8: Reusability of the catalyst

4.0 Conclusion

This research verified that the limestone used in the production of calcium oxide (CaO) catalysts for transesterification of various feedstocks to biodiesel is a very excellent resource which is in Okpella, Edo State, Nigeria. The general physicochemical characterisation indicates the existence of calcite ($CaCO_3$) in the original limestone with very few impurities, as shown by FTIR, XRD and TGA analysis. The process of calcification at 900°C was successful in converting $CaCO_3$ to crystalline and phase-pure CaO, which is very basic and highly catalytic. The XRF and EDS tests also showed that the concentration of CaO (89.23 wt.%) is also in agreement with other high-grade catalysts that are reported to occur in nature, such as in eggshells, mussel shells and marine limestone.

The morphological and textural analyses of the CaO produced showed that the material has a mesoporous structure, rough surface morphology and high surface area, which are all important characteristics for increased adsorption and catalytic reactions in the transesterification process. Experiments on reusability indicated that the catalyst was stable and could be used for six consecutive cycles with over 90% biodiesel yield, which was comparable to the behaviour of other naturally derived CaO catalysts. The findings support the use of Okpella limestone as the locally available and economically feasible raw material capable of maintaining a sustainable production of biodiesel, hence avoiding the use of imported catalysts to support a sustainable environment.

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